



# Development and Fatigue Testing of Ceramic Thermal Barrier Coatings

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Prepared for the  
International Conference on Metallurgical Coatings and Thin Films  
sponsored by the AVS Science and Technology Society  
Advanced Surface Engineering Division  
San Diego, California, April 19–23, 2004

National Aeronautics and  
Space Administration

Glenn Research Center

## Acknowledgments

The authors are grateful to George W. Leissler and Sandra L. Leissler, QSS Group, Inc., at the NASA Glenn Research Center, for their assistance in the preparations of plasma-sprayed  $\text{ZrO}_2$ -8wt% $\text{Y}_2\text{O}_3$  coatings and the metallographic specimens.

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# **Development and Thermal Fatigue Testing of Ceramic Thermal Barrier Coatings**

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## **Summary**

Ceramic thermal barrier coatings will play an increasingly important role in future gas turbine engines because of their ability to effectively protect the engine components and further raise engine temperatures. Durability of the coating systems remains a critical issue with the ever-increasing temperature requirements. Thermal conductivity increase and coating degradation due to sintering and phase changes are known to be detrimental to coating performance. There is a need to characterize the coating thermal fatigue behavior and temperature limit, in order to potentially take full advantage of the current coating capability. In this study, thermal conductivity and cyclic fatigue behaviors of plasma-sprayed  $\text{ZrO}_2\text{-}8\text{wt}\%\text{Y}_2\text{O}_3$  thermal barrier coatings were evaluated under high temperature, large thermal gradient and thermal cycling conditions. The coating degradation and failure processes were assessed by real-time monitoring of the coating thermal conductivity under the test conditions. The ceramic coating crack initiation and propagation driving forces and failure modes under the cyclic thermal loads will be discussed in light of the high temperature mechanical fatigue and fracture testing results.

## **Introduction**

Ceramic thermal barrier coatings (TBCs) have received increasing attention for advanced gas turbine engines. The future TBC systems will be more aggressively designed for the thermal protection of engine hot section components, thus allowing significant increase in engine operating temperatures, fuel efficiency and engine reliability. However, the increases in engine temperature and heat flux can raise considerable coating durability issues. The development of next generation advanced TBCs will greatly rely on the better understanding of the coating behavior and failure modes under the high temperature, high thermal gradient cyclic conditions.

Critical coating properties affecting the TBC life, such as ceramic sintering and creep, and thermal conductivity change kinetics at high temperatures, have been previously addressed (refs. 1 through 5). Fundamental TBC materials properties including deformation, cyclic fatigue, creep and fracture behavior have also recently been characterized (refs. 6 through 12). This present paper is primarily focused on thermal fatigue properties of plasma-sprayed TBC systems under temperature and stress gradients that can be encountered in advanced engine systems. In particular, quantitative crack propagation in a TBC

system is investigated under laser heat flux thermal cyclic loading. The coating life prediction methodologies are discussed based on the materials response and failure mechanisms observed under the high-heat-flux tests.

## **Experimental Materials and Methods**

Air plasma-sprayed (APS)  $\text{ZrO}_2$ -8wt% $\text{Y}_2\text{O}_3$  thermal barrier coatings were used for the coating thermal fatigue-crack propagation study. In this work, 25.4 mm-diameter and 3.2 mm-thick nickel base superalloy specimens were used as substrates. This button specimen configuration, which was previously used for coating thermal conductivity studies (refs. 5 and 13), is ideal for investigating the coating delamination crack propagation because the coating conductivity change is sensitive to the delamination cracks. The test coating system consists of an air plasma-sprayed  $\text{ZrO}_2$ -8wt% $\text{Y}_2\text{O}_3$  top coating (typical thickness range about 127 to 200  $\mu\text{m}$ ) and an intermediate low pressure plasma-sprayed NiCrAlY bond coat (thickness about 120  $\mu\text{m}$ ), plasma-sprayed on the button specimen substrates. A through-thickness hole, with either 1 or 2 mm diameter, was drilled in the center of the substrate by the electro-discharge machining (EDM). The central substrate hole thus prepared was able to successfully initiate a controlled coating delamination crack during initial laser thermal cyclic loadings.

A high power  $\text{CO}_2$  laser was used to test the TBC specimens under high temperature, high thermal gradient cyclic conditions, thus providing the necessary thermal fatigue loading for advancing the pre-initiated delamination cracks within the ceramic coatings near the ceramic/metal interface. A uniform laser power distribution was achieved over the specimen coating surface by using an integrating ZnSe lens combined with the specimen rotation. A description of the laser test rig system and the general approaches to testing the TBCs have been described elsewhere (refs. 4 and 5). For this work, low to medium heat fluxes of 60 to 100  $\text{W}/\text{cm}^2$  were used depending on the specimen coating thickness. Initial ceramic surface temperature was set at approximately 1287 °C. Either 10 or 20 min heating/cooling cycles were used for the laser thermal gradient cyclic tests. During the test, the ceramic surface temperature was measured by an 8  $\mu\text{m}$  infrared pyrometer. The back surface of the metal substrate was measured by both two-color and calibrated 8  $\mu\text{m}$  infrared pyrometers. The simultaneous measurements of the surface and backside temperatures provide transient heating temperature information, which is especially important for the cracked coating specimens. The spot size (diameter) of the 8  $\mu\text{m}$  pyrometer is 8 to 10 mm. The spatially weighted-average temperature information at coating surface in the presence of a delamination crack was calibrated in-situ with a high sensitivity camera under the given crack sizes, and ex-situ with a blackbody calibration furnace with various aperture sizes.

## **Experimental Results and Discussion**

### **Effective Thermal Conductivity and Delamination Crack Propagation**

The laser thermal fatigue and crack propagation study is based on a modified thermal conductivity test. In this test, the surface temperature and metal backside temperature of a pre-cracked TBC specimen are continuously measured during the laser cyclic thermal fatigue test. The nominal, or effective thermal conductivity of the ceramic coating can thus be determined as a function of the laser cycle number. The effective thermal conductivity at any given cycle contains valuable information about the advancing delamination crack in the coating in laser thermal cyclic loading. Figure 1 shows typical test results for a 127  $\mu\text{m}$  thick, precracked TBC specimen under the laser cyclic loading. It can be seen that the surface temperature increased continuously due to the crack initiation and propagation. The metal backside temperature and the predicted metal/ceramic interface temperature, however, remained relatively constant

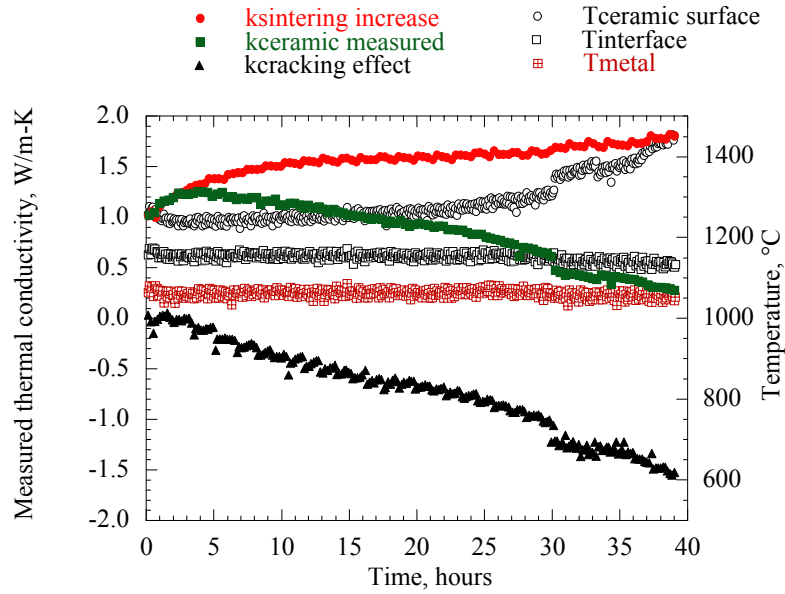


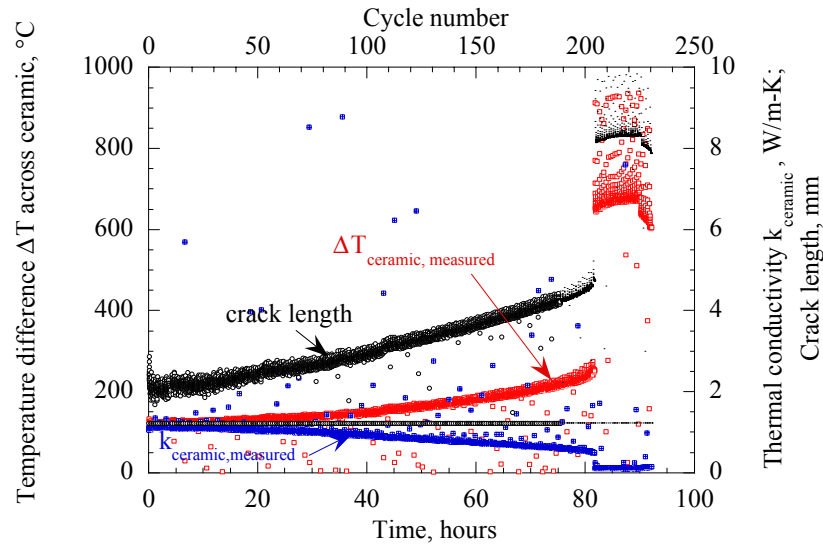
Figure 1.—Typical laser thermal fatigue test results of a 127  $\mu\text{m}$  thick, precracked TBC specimen showing the coating temperature and thermal conductivity changes as a function of cycle number under 10 min heating and 2 min cooling laser cycling. The ceramic surface temperature increases and the metal backside temperature slightly decreases as the delamination crack is initiated and propagated. The effective ceramic coating conductivity shows an initial increase due to the coating sintering, and then a decrease due to the crack propagation. A large conductivity drop during the test indicates

or exhibited a slight decreasing trend. The measured ceramic coating conductivity initially increased and decreased with further increasing the testing time and cycle number. The initial rise in the measured conductivity is attributed to the ceramic sintering effect. It is also noted that a sudden drop in thermal conductivity that is corresponding to a large surface temperature jump, was observed at about 30 hr, suggesting a coating spallation event occurred at that time.

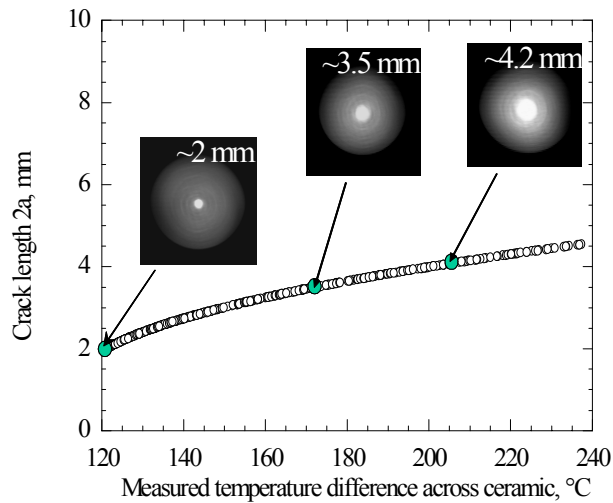
The measured ceramic thermal conductivity  $k_{\text{measured}}$  for a precracked TBC specimen can be expressed as

$$k_{\text{measured}} = k_{\text{intact}} + k_{\text{cracking}} \quad (1)$$

where  $k_{\text{intact}}$  is the thermal conductivity of non-cracked TBC specimen, which typically increases with time or cycle number due to the ceramic sintering effect, and  $k_{\text{cracking}}$  is the thermal conductivity reduction due to the coating cracking effect. The  $k_{\text{intact}}$  can be estimated from the previously reported coating property data under the current test conditions (refs. 3 and 14). From the  $k_{\text{intact}}$  and measured  $k_{\text{measured}}$  data, the  $k_{\text{cracking}}$  can be obtained, as shown in figure 1. The  $k_{\text{cracking}}$  showed a monotonic decreasing trend that corresponds to the delamination crack growth in the TBC system during laser thermal cycling. Figure 2 (a) shows similar test results of a TBC specimen with a 2 mm hole in the substrate. Again, the change in the specimen effective thermal conductivity clearly quantified the evolution of the crack propagation and coating spallation behavior. The coating propagation process has also been confirmed independently by a high sensitivity video camera, as shown in figure 2(b) (ref. 15).



(a)



(b)

Figure 2.—The laser thermal fatigue test results of a 0.2 mm thick TBC specimen with a 2 mm hole in the substrate when exposed to 20 min heating and 4 min cooling laser cycling. (a) A close relationship between the coating thermal conductivity and delamination crack length is demonstrated. The changes in the coating's effective thermal conductivity can be used to quantitatively describe the evolution of the delamination crack propagation and coating spallation behavior. (b) The coating propagation process has been monitored independently by a high sensitivity video camera, and expressed as a function of measured temperature difference across the coatings.

### TBC Buckling, Spallation and Critical Failure Strains

Under the relatively low heat flux laser thermal cycling of thin coatings, the coating failure mode was found to be primarily coating delamination crack growth with eventual buckling spallation. Figure 3 shows crack lengths and the corresponding crack propagation rates of several laser-tested TBC specimens



as a function of cycle number. For the specimen tested, the initial average crack propagation rate was in the range of 3 to 8  $\mu\text{m}/\text{cycle}$ . However, the crack propagation increased significantly with further laser cycling. At the later stage near the coating spallation, the crack propagation rates increased to as high as 30 to 40  $\mu\text{m}/\text{cycle}$ . A mixed coating delamination crack propagation path (either in the ceramic near the ceramic/bond coat interface or in the thermally grown oxide scales) was observed for the tested specimens. The accelerated crack growth under laser heat flux cyclic conditions is attributed to the increased driving force (the increased thermal stresses) for the crack propagation. A critical crack size can be reached to initiate catastrophic coating spallation from the subcritical crack propagation during laser thermal cyclic loading exposure.

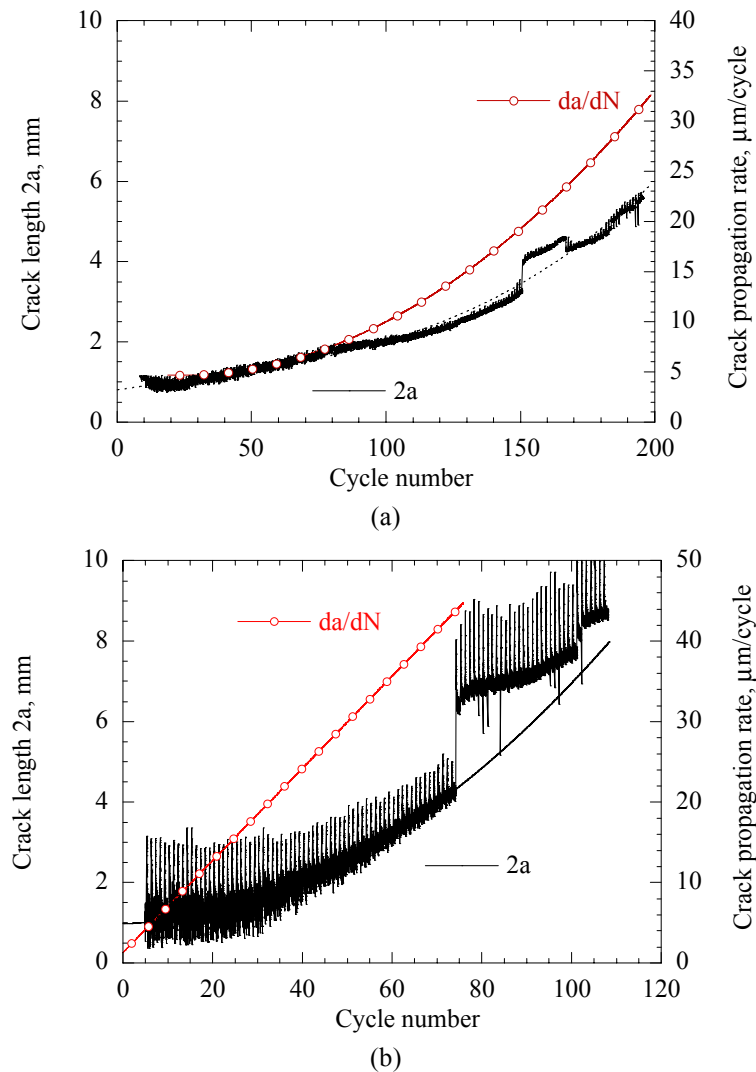


Figure 3.—Crack lengths and the corresponding crack propagation rates of laser tested TBC specimens as a function of cycle number. The crack propagation rates increase with the cycle number. The accelerated crack growth under laser heat flux cyclic conditions is attributed to the increased driving force for the crack propagation. (a) 127  $\mu\text{m}$  thick coating; (b) 176  $\mu\text{m}$  thick coating.

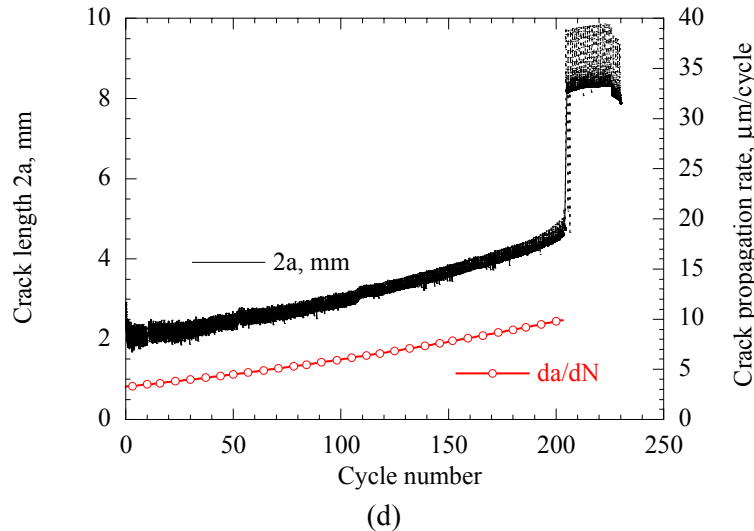
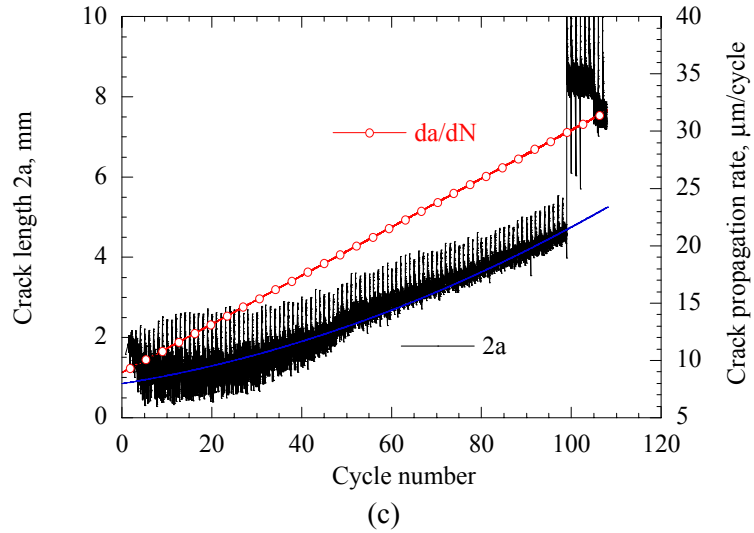


Figure 3.—(Concluded) Crack lengths and the corresponding crack propagation rates of laser tested TBC specimens as a function of cycle number. The crack propagation rates increase with the cycle number. The accelerated crack growth under laser heat flux cyclic conditions is attributed to the increased driving force for the crack propagation. (c) 185  $\mu\text{m}$  thick coating; (d) 200  $\mu\text{m}$  thick coating.

The critical buckling-spallation crack size,  $2a$  (the diameter of an approximate penny-shaped delamination crack), can be determined from the crack size plots (figure 3) at the coating spallation point. The critical crack size for initiating spallation was found to be in the range of 3 to 5 mm. However, the final spallation size for the coating ranged from 5 to 12 mm after the laser cyclic testing, due to the fast, unstable crack propagation after the initial spallation. The critical spallation size as a function of coating thickness was plotted in figure 4. It can be seen that a larger critical crack size is needed to spall a thicker coating. For simplicity purpose and without considering the complex nature of thermal visco-plasticity behavior of the TBC system, the critical coating buckling spallation strain can be estimated from the classical elastic theory for a clamped circular plate (ref. 16) as

$$\sigma_{spall} / E = \left[ \kappa / 12(1 - \nu^2) \right] (t / a)^2 \quad (2)$$

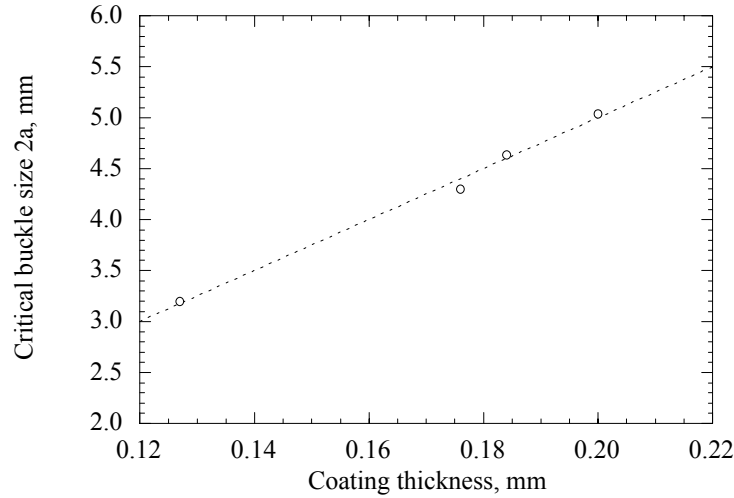


Figure 4.—The critical spallation size of the  $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$  coatings as a function of coating thickness under laser heat flux testing.

where  $\sigma_{spall}$  is the critical buckling/spallation stress,  $E$  and  $\nu$  are Young's modulus and Poisson's ratio of the coating, respectively,  $\kappa$  is a constant about 14.68,  $t$  is the coating thickness, and  $a$  is the crack radius at the spallation. The compressive, critical spallation strain is about 0.8 percent for the plasma-sprayed thermal barrier coatings under the laser test conditions.

### Fatigue Crack Subcritical Growth

The delamination crack propagation rate  $da/dN$  under the laser thermal cyclic loads can be generally expressed as

$$da/dN = da/dN_{heating} + da/dN_{cooling} = C_1 \Delta K_{heating}^m + C_2 \Delta K_{cooling}^m \quad (3)$$

where  $da/dN_{heating}$  and  $da/dN_{cooling}$  are the crack propagation rates under laser thermal transient heating and cooling, respectively,  $\Delta K_{heating}$  and  $\Delta K_{cooling}$  are the stress intensity factor amplitudes associated with the heating and cooling thermal loads,  $C_1$ ,  $C_2$ , and  $m$  are constants. Under the laser heat flux test conditions, due to the relatively large temperature difference ( $\Delta T$ ) across the ceramic coating, the residual compressive stresses for driving the coating delamination crack propagation are greatly reduced as compared to the uniform heating-cooling case. On the other hand, the dynamic transient thermal stresses, induced during the laser surface rapid heating of the partially delaminated coating may become a more predominant driving force for the crack propagation, primarily because of the large thermal compressive stress magnitudes and the fast dynamic loading characteristics near the crack tip. Previous dynamic fatigue tests of the plasma-sprayed  $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$  have shown that the coating fracture stress is significantly reduced with increasing the loading stress rate. This phenomenon has been attributed to the reduced crack tip visco-plasticity induced blunting under the high stress rates (ref. 11).

In this study, in order to assess the effect of the thermal transient stress intensity factor amplitude  $\Delta K$  on coating crack propagation, a coating delamination and buckling spalling model was used (ref. 17). Based on the stress amplitude derived from the measured transient temperature profiles of the coating system, the subcritical crack propagation driving force  $\Delta K$  can be estimated as

$$\Delta K = [(1 - \alpha)/(1 + \nu)]^{1/2} [1 - (\sigma_c / \Delta \sigma_t)^2]^{1/2} \Delta \sigma_t t^{1/2} \quad (4)$$

where  $\alpha$  is a constant and  $\alpha = 0.399$  if taking  $\nu = 0.25$ ,  $\Delta \sigma_t$  is the transient stress associated stress amplitude,  $\sigma_c$  is the post-buckling stress in the coating. In general,  $\sigma_c \ll \Delta \sigma_t$  under the laser high temperature test conditions. Note that the stress intensity factor amplitude,  $\Delta K$ , increases with laser thermal cycle number, because of the increased temperature difference  $\Delta \sigma_t$  with increasing coating crack length  $2a$ . Figure 5 illustrates the relationship between the delamination crack propagation rate  $da/dN$  and the laser thermal transient stress associated stress intensity factor amplitude  $\Delta K$ , during the laser thermal fatigue testing. The exponent,  $m$ , for the delamination fatigue crack growth under the laser thermal cycle driving force  $\Delta K$ , was found to be about 5. Slightly lower  $m$  values were observed under the larger  $\Delta K$  conditions, which may be related to the complexity of the coating delamination-buckling mechanism under the laser thermal gradient testing. Figure 6 shows micrographs of laser thermal fatigue tested TBC specimens. As shown in figure 6(a), severe fatigue damages are observed near the early crack propagation wake surfaces with strong coating asperity/debris interactions and coating multiple delaminations under the laser thermal cyclic loading. However, the later crack paths show relatively smooth surfaces, which corresponds to the faster crack propagation regions under the increased crack propagation driving force. A coating spallation morphology after the laser thermal fatigue test is shown in figure 6(b).

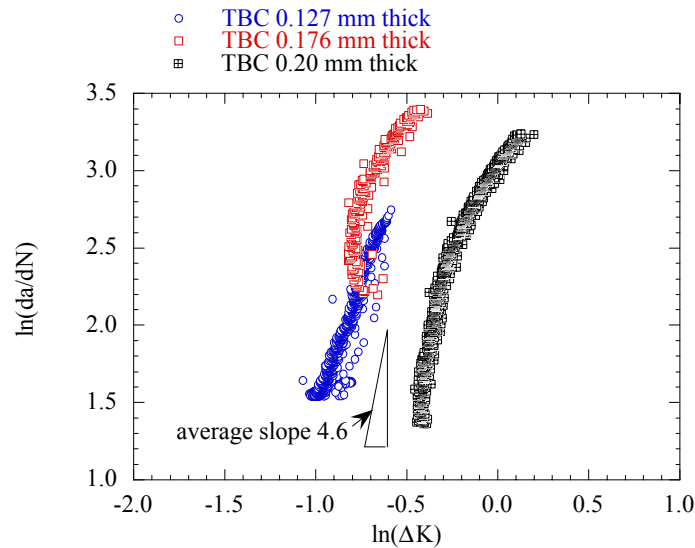


Figure 5.—The relationship between the delamination crack propagation rate  $da/dN$  and the laser thermal transient stress associated stress intensity factor amplitude  $\Delta K$ .

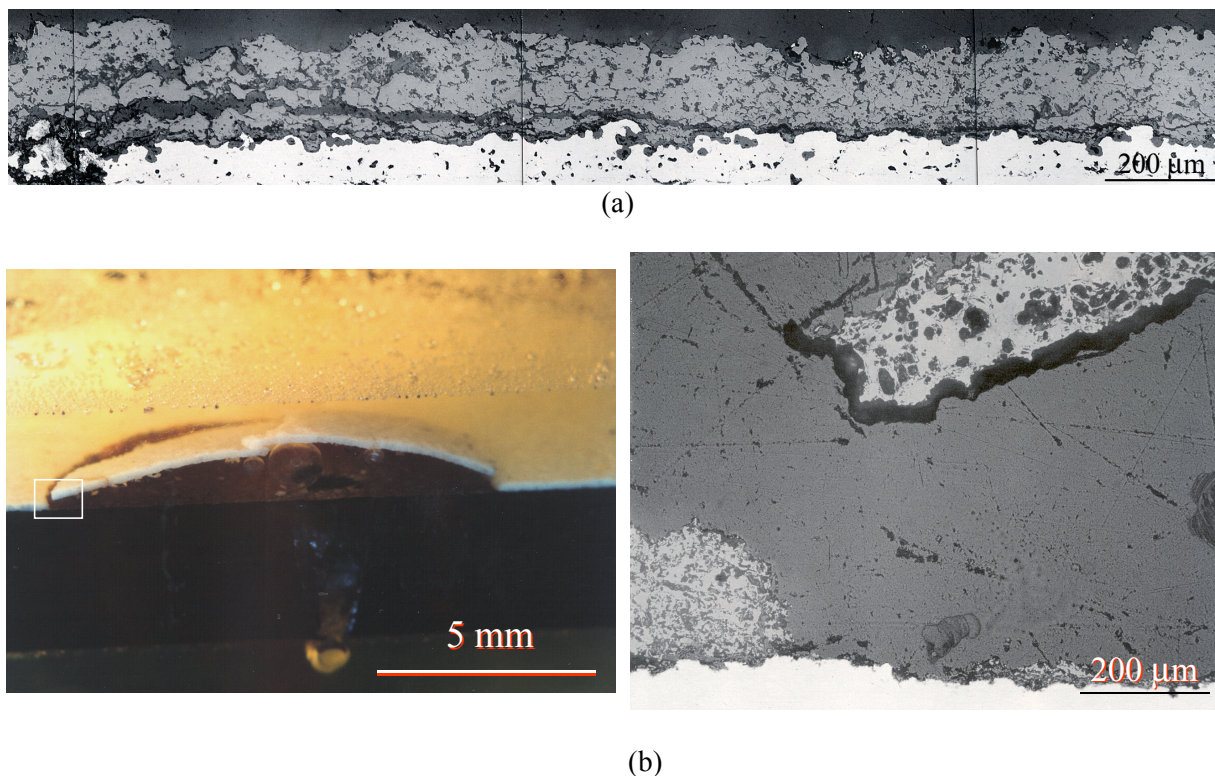


Figure 6.—Micrographs of laser thermal fatigue tested TBCs showing the coating delamination crack propagation and coating spallation. (a) Severe fatigue damages are observed near the early crack propagation wake surfaces with strong coating asperity/debris interactions and coating multiple delaminations under the laser thermal cyclic loading. The later crack paths show relatively smooth surfaces, which corresponds to the faster crack propagation regions under the increased crack propagation driving force. (b) Coating spallation morphology after the laser thermal fatigue test.

## Conclusions

A laser thermal fatigue test technique has been used to study the delamination crack propagation of thermal barrier coatings under the heat flux heating and thermal cyclic loading. For the  $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$  coating specimen tested, the initial average crack propagation rate was in the range of 3 to 8  $\mu\text{m}/\text{cycle}$ . The crack propagation rates increased to 30 to 40  $\mu\text{m}/\text{cycle}$  at the later stage of the tests. The coating spallation failure mode was observed, and the critical spalling crack size ranged from 3 to 5 mm for the TBC coatings. The accelerated crack growth is attributed to the increased driving force for the crack propagation under the laser heat flux cyclic test conditions.

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE July 2004		3. REPORT TYPE AND DATES COVERED Technical Memorandum
4. TITLE AND SUBTITLE  Development and Fatigue Testing of Ceramic Thermal Barrier Coatings			5. FUNDING NUMBERS  WBS-22-714-20-09 1L162211A47A	
6. AUTHOR(S)  Dongming Zhu, Sung R. Choi, and Robert A. Miller				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)  National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER  E-14561	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)  National Aeronautics and Space Administration Washington, DC 20546-0001 and U.S. Army Research Laboratory Adelphi, Maryland 20783-1145			10. SPONSORING/MONITORING AGENCY REPORT NUMBER  NASA TM-2004-213083 ARL-TR-3261	
11. SUPPLEMENTARY NOTES Prepared for the International Conference on Metallurgical Coatings and Thin Films sponsored by the AVS Science and Technology Society, Advanced Surface Engineering Division, San Diego, California, April 19-23, 2004. Dongming Zhu, U.S. Army Research Laboratory, NASA Glenn Research Center; Sung R. Choi, Ohio Aerospace Institute, 22800 Cedar Point Road, Brook Park, Ohio 44142; and Robert A. Miller, NASA Glenn Research Center. Responsible person, Dongming Zhu, organization code 5160, 216-433-5422.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT  Unclassified - Unlimited Subject Categories: 23 and 26  Available electronically at <a href="http://gltrs.grc.nasa.gov">http://gltrs.grc.nasa.gov</a> This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
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14. SUBJECT TERMS  Thermal barrier coatings; Thermal fatigue testing; Laser heat flux; Thermal conductivity			15. NUMBER OF PAGES 16	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT  Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE  Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT  Unclassified	20. LIMITATION OF ABSTRACT	





